#### APPENDIX D

# COMMENTS ON THE U.S. ENVIRONMENTAL PROTECTION AGENCY'S PROPOSED MONITORING METHOD FOR FINE PARTICULATE MATTER

#### **SUMMARY**

One of the technical challenges to support implementation of specific standards for fine particulates is the need for a federally-approved air monitoring method and strategy. While there are some technical and implementation issues with the U.S. Environmental Protection Agency (U.S. EPA) proposals for monitoring PM2.5, these questions can and should be addressed expeditiously. U.S. EPA should work with state and local agencies to craft creative solutions, then provide the funds needed to implement them.

As proposed, the PM2.5 federal reference method will not adequately identify the two most common constituents of fine particulate matter in California -- ammonium nitrate and carbon. In some California cities, ammonium nitrate can represent over 60 percent of the total PM2.5. Accurate quantification and characterization of the fine particulate problem is essential to the development of appropriate and cost-effective controls. *U.S. EPA should modify its* proposed procedures, particularly those related to filter substrate and filter handling, to improve the accuracy of the measurement technique.

U.S. EPA will not have completed field testing to demonstrate and refine the proposed sampling methods prior to the scheduled final approval in July 1997. Instead, the agency proposes to shift the responsibility and risk to the states. If state operation of an "approved" monitor subsequently indicates that the results are not consistent with the federal reference method, U.S. EPA would rescind approval of the technology, regardless of the state's investment in equipment and operation of the monitors. *U.S. EPA should assume full responsibility for the field testing and validate the monitors before prescribing specific technology and methods.* 

This appendix is organized into three sections. In the first section, we present our comments on the proposed federal reference method itself. Many of these comments relate to our concern that the method does not adequately protect against the loss of volatile species. We offer recommendations to improve the method's performance. In the second section, we present our comments on the field testing of PM2.5 monitors. Finally, in the third section, we present additional comments, recommendations, and questions on the specific proposed regulatory text of Part 50 Appendix L--Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere.

#### FEDERAL REFERENCE METHOD FOR PM2.5

## **Volatile Species Losses**

The loss of volatile species from filter-based sampling is well documented and understood. Losses from PM10 filters can be significant and are suspected to occur during active sampling, while the filter resides on the sampler, during transportation to the laboratory, and during storage and equilibration of the filter itself. Ammonium nitrate, a very volatile species, evaporates during these phases of the sampling process. Volatile organic species are similarly affected. The result is that these volatile species are regularly and substantially underrepresented. Nitrates and carbon particulates are both leading components of PM2.5. Unless the suggested improvements are made to the proposed federal reference method (FRM), it would likely underestimate the contribution of volatile compounds to PM2.5 levels, which could result in ineffective PM plans, strategies and controls.

U.S. EPA staff has suggested that the volatile species sampling problem could be mitigated by funding development of a variety of non-FRM and research grade instruments. This would help, as experimental samplers could be designed to retain volatile species better than the FRM. Regulatory decisions, however, specifically those related to mass measurements, would continue to be based on data obtained from the FRM. Based on the degree of difference, accurate species data from a non-FRM device would not be comparable to the same species determined from the FRM, nor would their mass values. The result would be to create dissimilar air quality data sets that would track the effect of new control measures differently. Simply stated, one cannot effectively control what one cannot adequately measure.

U.S. EPA must make the necessary changes to make the FRM a reliable method for both mass and species determinations. Please see an attached copy of a letter we sent to U.S. EPA, dated June 1996, in which we suggest an addition to the proposed FRM that attempts to address the nitrate question. We also suggest several practices, primarily related to filter handling/processing, that will reduce constituent losses and establish greater uniformity in measured values across the country. Furthermore, proper filter handling will improve overall accuracy of the mass measurement from the proposed FRM. Specific comments related to the FRM and the volatile species issues follow.

We suggest U.S. EPA closely examine reducing volatile losses, at a minimum, during the period after sampling and before extraction/weighings. Significant losses occur during this time and relatively simple improvements could be made now. We are examining the effect of volatile losses actually during equilibration, but have no information available to report at this time.

The proposal attempts to reduce volatile losses in several ways: temperature control, reduced post sampling time in the field, and expedited transport and post weights by the lab.

## **Post Sampling Filter Handling**

We suggest that filters be removed from the sampler as soon as is practicable (no later than 24-48 hrs) and immediately placed in a cool container. Preliminary results from a California Air Resources Board study in progress in Bakersfield, California, show that nitrate levels from Teflon filters/low volume samplers received at the lab are comparable to those obtained from onsite extractions, provided the sample filter was placed immediately on ice.

Additionally, we have seen little degradation in nitrate readings thus far when the filters are maintained on ice for two days or seven days. One possible outcome of the study may be that filters placed in a controlled environment may not need to be received by the lab within 96 hours as the regulation would require.

## **Temperature Control On-Site**

We appreciate the need to minimize the excessive heat build-up in the sampler. However, we question the need to track the temperature difference between the ambient and filter ( $\triangle T$ ; deg C) for each sample. We assume U.S. EPA would require this criteria of the vendor and require tests under controlled conditions prior to granting reference status. If done properly, the confirmatory testing would negate the need for tracking  $\triangle T$  at all. It would reduce the operator's burden and eliminate the potential for deleting potentially useful data (see comments on Section 7.4.11 below).

There seems to be little assurance that the  $\triangle T$  of the sample day will be maintained on subsequent days if the ambient temperature on those days increased moderately (e.g.,  $\pm 10$  deg F). If it is, filters will need to be kept in a temperature controlled environment on those days. If not, filters could reach temperatures greater than 3 deg C of the sample day's ambient temperature on those days. This would make the need to track on the sample day meaningless. Therefore, U.S. EPA should examine whether the sampler needs to be environmentally controlled. If not, U.S. EPA should determine whether tracking  $\triangle T$  is relevant, or if the goal of minimizing heat build-up in the sampler can be assured simply through early confirmatory reference testing that meets U.S. EPA's satisfaction.

Many air monitoring locations are already equipped with sophisticated ambient temperature and pressure instrumentation. Many are maintained to meet the strict quality assurance (QA) standards of the U.S. EPA's handbook for meteorological monitoring. It is unclear whether the proposed FRM meteorological sensors will be of comparable quality or provide comparable readings. Specifically, if the  $\Delta T$  requirement is maintained, the accuracy of the ambient measurement will be critical.

## Filter Media

Recent, extensive air quality studies in the South Coast Air Basin showed the leading components of PM2.5 on days of high concentration were almost entirely cations, anions, and organic material. In contrast to PM10, the crustal material fraction of PM2.5, commonly traced to crustal materials, was near trace level. Analyses of cations, anions, and organics are successfully done on quartz fiber filters. The proposed PM2.5 reference method specifies Teflon as the filter media. While Teflon is a suitable substrate for cations and anions, it does not permit analyses for organics. Given the potentially decreased need for elemental analyses, U.S. EPA should include a filter substrate option *in the reference method* capable of providing mass, ions and carbon. This would greatly facilitate analyses of the most abundant species and minimize the cost of second samplers to collect the needed information. The user should have the option of using either Teflon or an appropriately designed quartz filter *completely interchangeably*. This would extend to *all* regulations and cause the reference sampler to be entirely filter neutral with respect to the use of Teflon or quartz filters.

## **Design Concern**

It was noted that the separator chamber in the proposed PM2.5 FRM contains a glass fiber filter that is coated with non-polar silicone fluid. It has been our experience that the PM10 inlet used in the sampler may allow large amounts of water vapor to collect. If, during the separation of the PM10 and PM2.5 fraction, water is condensed on the silicone oil surface, a thin layer of water could form on top of the oiled filter. This film of water could then act as a collection device (denuder) for water soluble particles, such as ammonium nitrate and sulfuric acid. Since ammonium nitrate constitutes the major fraction of PM2.5 in winter months in California, and since fog is common during the winter months, it can be expected this design feature will cause the PM2.5 mass collected to be significantly less than ambient concentrations.

## Specific Language: 'Should v. Shall'

The language of previous air monitoring regulations, including PM10, has at times not clearly delineated the *advisory* from the *required* parameters for obtaining a valid measurement. Some practices and procedures are essential and bear directly on the accuracy of the resulting sample concentration. Others may be needed to establish national uniformity among the measured values. However, some items proposed in the regulation would be classified as advisory or as guidance and not significant to the integrity of the measurement itself. We request that the monitoring regulation in Parts 50, 53 and 58 include only those items U.S. EPA deems essential to the value being considered data for record (useable for all regulatory findings). U.S. EPA would have technical justification readily available to the states to support their position. The essential activities would be identified with the terms *must or shall*. We suggest that guidance-type statements, goals or targets be identified clearly by signifying them with the terms *should or may*. Perhaps the clearest distinction would be to retain only those items in the regulation that are critical to the measurement, and include in U.S. EPA's QA Manual the more

discretionary procedures and practices. To ensure consistent measurement from county to county, we would, as has been done for PM10, audit the field and lab portions of the program using standards traceable as required in the regulation by U.S. EPA. We would intend to take further action if samplers/labs did not comply with the critical elements.

## **Operating Requirements for Class I and II Equivalent Samplers**

Some of the sampler's operational practices proposed for the FRM also need to be incorporated into U.S. EPA's *equivalent method requirements*. Given the need to go beyond the FRM and develop sequential samplers, we expect relatively few field samplers will be of the reference design. By definition, innovative samplers will most likely be designated equivalent method instruments. Unless filter care, as proposed for the FRM, is made a requirement of equivalent methods, the utility of data from equivalent methods will be greatly diminished. U.S. EPA has an opportunity to improve the quality and uniformity of the PM2.5 data in a very substantial way. It is important, therefore, that any feature, policy, guideline, or practice embodied in the FRM that preserves the quality of sampling data, including volatile constituents, be made direct requirements of Class I and II equivalent methods and stressed with vendors of Class III samplers.

## FIELD TESTING OF PM2.5 MONITORS

The proposal would have states initiate, for the first time for any FRM, a new, comprehensive layer of field-testing requirements for PM2.5 samplers that had already been approved by U.S. EPA. These requirements appear in Part 50 and Part 58 and should be eliminated from the proposal.

We believe U.S. EPA has the obligation to conduct comprehensive evaluations of candidate reference and equivalent samplers before issuing its official approval of a sampler. Leaving open the question of sampler acceptance will have repercussions for sampler purchases, and possibly place regulatory decisions in jeopardy.

We continue to support a state's ability to assist vendors or U.S. EPA with field testing, but that involvement should remain voluntary. Clearly, the regulations must provide a measure of instrument performance, but not in the manner proposed. We believe the state's role is primarily to operate U.S. EPA approved samplers, including collocated samplers, and to ensure they remain within precision and accuracy limits established by U.S. EPA.

In a most unusual move, confirmatory testing, called a test of concordance, would be extended to the single reference method itself. U.S. EPA states that the accuracy of PM2.5 samplers cannot be established in an absolute sense. Instead, accuracy is established in a relative sense through the designation of a design-based sampling technique (i.e., the reference method).

The accuracy of the reference method is defined simply by technique selected to measure the particle matter.

What then is the U.S. EPA's rationale for defining the FRM sampler's accuracy relative to a measurement principle only to require every reference device to be compared against another reference device each year? By definition, the two samplers are based on the same measurement principle and have been shown to U.S. EPA's satisfaction to be comparable based on earlier field testing. A state's role, therefore, as indicated above, should be to verify that samplers are operated properly using conventional means of collocated sampling for precision and flow audits for accuracy. It is wholly inappropriate for state and local agencies to be asked to conduct ongoing, fundamental, research-level testing on samplers that are the foundation of critical decisions. To restate our view, U.S. EPA has the obligation to approve samplers suitable to the task.

The requirements for six tests to be conducted for several days at each site each year (the test of concordance) would be *in addition* to the U.S. EPA's proposed requirement for separate quarterly audits of specific operational parameters. Consequently, each sampler would be saturated with 10 distinct audits each year. This level of effort would consume at least 16 days, or fully one-quarter of the days the sampler would normally be collecting samples (assuming a non-core site).

The practice of reconfirming a U.S. EPA-approved reference sampler against another approved reference sampler is unprecedented. What confidence can one place in any reference method if one must continuously re-validate all reference samplers? We are uncertain whether U.S. EPA would use the data, as it is proposing, to rescind a FRM designation for poor precision or accuracy. One can only imagine the confusion it could create, especially after states had already purchased the FRM sampler and used its data for regulatory decision making. We suggest U.S. EPA obtain its needed quality control information using more conventional means.

## **Equivalency Testing**

We suggest U.S. EPA's equivalency testing program be expanded to give assurance of equivalency at or near the level of the standard, and also on high concentration days. It is increasingly important for the samplers to be linear throughout the expected range of concentrations. Attainment decisions require very comparable results, but so do other decisions that depend on high values, such as designations, categories of exceedances, design values, and trends. The expansion should include specific requirements to assure high values are adequately included in the testing program and specific analytical tests on high values to assure equivalency under conditions characteristic of high concentrations. Current federal equivalency comparison tests evaluate a wide range of data magnitudes, and allow for significant differences between how methods respond to high values, which can be critical in a regulatory context.

Moreover, equivalency testing needs to be conducted in a variety of real world conditions known to affect the sampler. Instrument response can be dependent on the particle composition. The composition can vary during times of the year and also over time as controls are implemented.

#### COMMENTS ON SPECIFIC SECTIONS OF PART 50 APPENDIX L

In this section, we present our concerns, questions, and recommendations regarding the proposed regulatory text of Part 50 Appendix L--Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere. Our comments are organized by section number in Part 50 Appendix L.

## **Section 3**

Section 3.3

This section was apparently added to ensure that samples whose collection time did not meet the requisite number of hours (23-25 hrs) could, under specific conditions, be considered a legitimate exceedance of the standard. The language states that the "measurement may be used as a valid concentration measurement for purposes of determining violations of the NAAQS." We suggest this could be construed as allowing *all measurements* to use this technique. The language needs to be tightened to ensure the proposed calculation would not allow any sample to be purposefully terminated early.

For samples that end early due to low flows, the sampler must be designed to note the time that the sample ran.

## Section 4

Section 4.1

Section 4.2.1 - 4.2.2

See comments above under **FIELD TESTING OF PM2.5 MONITORS**.

#### Section 4.3:

Part 58, Appendix A of this chapter requires that the flow rate accuracy of PM2.5 samplers used in SLAMS monitoring networks be assessed periodically via audits of the sampler's operational flow rate. We recommend annual audits as is the case with PM10.

It appears that temperature and barometric pressure will be critical items. If so, a check of the temperature (ambient and filter) and barometric pressure sensors should also be conducted along with the annual flow rate accuracy audits.

#### **Section 5**

Section 5.2.2

The examination for excessive imprecision should be done during periods of high particle concentrations. The 15% limit could be exceeded at very low (10-20 ug/m3) concentrations. Low level loadings should be exempted from the 15% limit.

#### **Section 6**

Section 6.8

Filter weight efficiency, states: Filter weight loss  $\leq$  20 ug, measured as specified in the following two tests. How does one do "initial and final weights . . . prior to shipment?" Are all of the filter requirements going to be tested by the manufacturer before they are shipped? What is the basis for the 20 ug criteria? Who is responsible for doing the tests -- U.S. EPA? the vendor? The regulation does not specify the action to be taken if the filters do not pass the tests, e.g., retest, reject the lot number, reject individual filters?

Section 6.9

Who is responsible for doing the alkalinity tests -- U.S. EPA? the vendor?

#### **Section 7**

Section 7.4.1

The flow specification is 16.67 +/- 0.83 lpm. Is there any work that shows this flow rate maintains the PM2.5 cutpoint for ambient temperature and pressure at elevations greater than 1000 feet? U.S. EPA should require the vendors to provide test data showing they meet the flow specification at elevated sites.

#### Sections 7.4.3, 7.4.4, and 7.4.5

This section implies that the sampler shall have a microprocessor controlled flow controller and flow measurement system. The flow measurement system needs to be continuous (mass flow sensor?) and needs to be stored in memory as well as having limit "switches" for "flags" or shutting down the sampler. Some sort of reporting device (printer, recorder, datalogger) would also be necessary, but is not specified. The system shuts down the sampling based on a 10 percent flow deviation for more than 60 seconds, but allows for the collection of a sample in case of a power outage. This doesn't make sense.

What statistical level of data completeness is U.S. EPA seeking if a sample is to be stopped for the day if it deviates from the sample flow rate by 10 percent for less than one percent of the 24 hour run? This level of data completeness is unusual and needs to be justified because of the potential for loss of otherwise representative data.

The sampler shall provide a means to measure and display the instantaneous sample air flow, which shall be measured as volumetric flow at the temperature and pressure of the sample air entering the impactor, with an accuracy of  $\pm$ two percent. Is  $\pm$ two percent intended to be used as a control limit for flow rate accuracy audits? Has accuracy at this level been demonstrated?

#### Section 7.4.6

This section would require a built-in vacuum measurement device capable of reading 112 +/- 10 mm Hg and a difference of 10 mm Hg. It also implies, but does not specify a recording or reporting device. This could be a vacuum gauge attached to the sampler, but it would have to be a good quality gauge and the gauge would require auditing.

#### Section 7.4.7

The sampler would be required to operate properly within an ambient temperature range of -20 to 40 deg C. If a sampler is in the direct sun, wouldn't the filter regularly exceed the "ambient temperature" and the proposed requirement for temperature differences between the filter and ambient air? Will samplers need to be shielded from the direct sun?

Ambient temperature in California exceeds 40 deg C in the summer in several areas. Similarly, the minimum barometric pressure of 600 mb would be inadequate in high mountain areas in California. Both limits need to be reviewed. How can the required range for ambient *operation* (-30 to +45 deg C) be wider than the required range for *testing* (-20 to +40 deg C)?

The regulation would require that ambient temperature, filter temperature, and barometric pressure be measured continuously at all times. It is implied that this measurement would be made to "effect filter temperature control." How would this be done? Also, some sort of recording device will be necessary, although it is not specified.

#### Section 7.4.9

This section states, "... barometric pressure measurement may be used for purposes of computation of volumetric flow rates, if appropriate." Isn't this in contradiction of Section 2.2?

#### Section 7.4.11

The second sentence in filter temperature sensors states: "The sampler shall also provide a warning flag indication following any occurrence in which the filter temperature exceeds the ambient temperature by more than 3 deg C for more than 10 consecutive minutes . . ."

Are filters that exceed ambient temperature by 3 deg C (5.5 deg F) for 10 minutes valid samples? At what point do the samples become invalid? Would a sample whose filter reached 80 deg F on a day the ambient temperature was 72 deg F (outside the 3 deg C limit) be any less valid than one whose filter temperature reached 102 deg F when the ambient temperature reached 104 deg F (within the 3 deg C limit)?

Must sequential samplers maintain the 3 deg C limit below ambient temperature for each sample based on each day's temperature, for the most recent sample only, or for the one collected at the lowest ambient temperature?

#### **Section 7.4.15**

This section implies the need for uninterruptible power supply (UPS), not just a back-up battery. The battery needs to be big enough to supply power to clock and microprocessor for seven days.

#### **Section 7.4.17**

A simple, built-in printer, not just the RS-232 output, should be required so the operator can review the operating parameters easily and attach the printout to the record. We expect some samplers will be found at sites without data loggers and information about the run would be lost unless the site operator had a means of printing it out. The printout would also be particularly valuable for those not on site, i.e., laboratory staff or others needing to do quality control (QC) on the sampler at a remote site.

#### Section 7.4.19

Table L-1 on pages 20 and 21 contains a summary of information to be provided by the sampler. We suggest U.S. EPA carefully review the list of operating parameters and only require those essential to the measured value. Collecting, transcribing, assessing, possible key-entering voluminous amounts of information will add a significant burden to the site operator. It is essential that each parameter be absolutely necessary to justify this level of increased work. We strongly urge U.S. EPA to eliminate all non-essential requirements and simplify the data gathering and handling of the day-to-day operations.

The AIRS reporting requirements for sampler QC data (those indicated by a • on Table L-1 in the proposal), are unnecessary and unjustified. The regulations would require site operators not just to review and approve, but also *submit to AIRS* detailed and extensive QC information *for every sample run*. There is no rationale given for this dramatic increase in U.S. EPA's direct oversight of state and local monitoring. Current PM10 data reporting simply includes the sampling date, site identification, and particulate mass concentration. Collocated sample data are included to assess system precision. This proposal, however, would have states submit that information to AIRS, *but also*:

- the coefficient of variation of the five minute averaged flow rate values of the sample period;
- the five minute average out of spec data and check flags;
- the sample volume;
- ambient temperature (maximum, minimum, and average) for the sample period;
- ambient barometric pressure (maximum, minimum, and average) for the sample period;
- instantaneous filter temperature plus check flags;
- sample start time; and
- elapsed sample time, and check flags.

U.S. EPA needs to justify its need for such detailed data for each site and each sample. It is an appropriate responsibility for the station operators to ensure sample validity considering the QC

information available. However, these data need not be submitted to AIRS quarterly. The data reporting requirements should be the same as those for PM10.

## **Section 8-Analysis**

Section 8.1

Analytical balance states: The balance shall be calibrated as specified by the manufacturer at installation and recalibrated immediately before each weighing session, but not less than once per year.

Does calibration refer to annual balance calibration and recalibration to daily balance calibration using a set of standard weights?

Balance calibration requirement for PM2.5 should be the same as for PM10. With PM10, standard weight checks (daily calibrations) are conducted each day of filter weighing using a set of standard weights and an annual calibration of the balance is conducted according to the manufacturer's recommendations. This should be explained more clearly here or in Section 2.12.

The weight range for the daily calibration should be specified along with a statement that the weights used for the calibration should (or must) be NIST traceable.

#### Section 8.2

The filter conditioning/equilibration requirements are:

8.2.1 Mean Temperature: 20-23 deg C

8.2.3 Mean humidity: 30-40 percent relative humidity

Is there a scientifically justifiable basis for the temperature range requirement? How were these requirements determined? What is the justification for having different equilibration temperature for PM2.5 and PM10? What is the reference study to show variability in filter and/or sample mass with temperature? Is U.S. EPA going to support data invalidation if these requirements are not satisfied?

#### Section 8.3.1

Sample filters should be weighed as soon as possible after arrival in the lab; however, if filters are shipped and temporarily stored on ice, there may be less need to equilibrate the filters immediately upon receipt at the lab.

#### Section 8.3.2

This section would require a dedicated environment-controlled weighing room for filter analysis. Many labs doing PM10 work are conditioning filters in small chambers and weighing them immediately upon removal under normal conditions. Is there significant benefit to this more stringent requirement over that of PM10? A recent addition to our facilities for a new environment-controlled room cost \$20,000 (less balance). Will U.S. EPA be providing funding

for small districts to build up their lab capabilities? This single requirement would likely make it impossible for small districts (many non-Section 105 grant recipients) to conduct their own sample weighings.

#### Section 8.3.4

This section would require that pre- and post- filter weight *must be carried out by the same analyst on the same balance*. This section should be eliminated as it is contradictory to our experience and the notion of established quality control. It assumes significant differences between laboratory equipment and the abilities of trained staff that simply does not exist. The proposal would cause filters to be left unweighed if an analyst was unavailable to do both ends of the weighings.

#### Section 8.3.5

The pre-sampling (tare) weighing shall be within 30 days of the sampling period.

This requirement would pose a burden on centralized laboratories that deliver preweighed filters to many sites quarterly. Assuming the filters will be stored in closed containers, the tare weight should remain stable for an extended period. The time needs to be defined as "within a quarter" unless U.S. EPA has evidence to the contrary.

#### Section 8.3.6

This section would place a time limit on the interval between the conclusion of sampling and filter weighings. This is an important component of the measurement process if we are to conserve mass on the filter. However, given the number of individuals/organizations involved in the filter handling process, especially in a centralized laboratory, samples could be invalidated if the limit was established at 240 hours. If proper precautions are taken, i.e., the filters are placed in a container and stored immediately on ice, the time to post weighings could be relaxed.

#### Section 8.3.7

This section needs clarification. Is the trip-blank filter to be sent to and from the sampling site or is it to be placed in a sampler (not run) and retrieved to be sent back for reweighing? The number, or percent, or frequency of 'blank' filters needs to be stated.

#### Section 9

#### Section 9.2.2

The flow rate standard must be re-calibrated or re-verified at least annually.

Does this mean that if a re-verification is within limits, at least annually, that a re-calibration (multi-point) is not required annually?

#### Section 9.2.4

The "at least five separate flow rate measurements (multi-point calibration) . . . " statement is excessive. A single point above the desired sampling flow rate, one below and one at the 16.7 should be adequate.

#### Section 9.2.5

The  $\pm -4\%$  verification seems too extreme. A  $\pm -7\%$  level seems more appropriate.

#### Section 9.2.6

The sampler should be calibrated with clean filters in place before the transfer standard is removed. The sampler's normal flow rate (in L/min) would be set at 16.7 at that point.

#### **Section 10**

#### Section 10.7

The regulation would require the filter holders to be made of metal. What is the rationale for metal as opposed to plastic? The current sample filter holders used in the current nitrate loss study at Bakersfield have shown consistent results between samples extracted on site and those shipped and aged in plastic shipping containers.

#### Section 10.13

The filter should be maintained on ice after removal and not be allowed to reach 32 deg C anytime.

#### Section 10.17

There is clearly a need for samples to be speciated to further our understanding of particulate constituents that comprise the mass and to comprehend better the effect of these components. We see little value, however, in requiring speciation in the regulation and believe the frequency of chemical analyses should be left up to the states. However, we support there being a greater awareness for actively preserving filters that may be speciated either immediately or later. In previous comments to U.S. EPA staff, we suggested filters conditions needed to be specified if one expected to use archived filters to produce reliable results. Our experience with refrigerated dry acid deposition samples extracts (Teflon), however, is that they degrade in 6-9 months. Whole filters stored frozen showed significant degradation after 18 months or less. Consequently, one should analyze filters relatively soon after sampling. Archiving filters looks promising only to a point. The regulation should discourage prolonged archiving unless the integrity of the filters can be scientifically assured.

## OTHER PARTS

## Part 58, Appendix A, Section 3.2.2.

Our experience with flow audits of PM10 and other manual samplers does not support the need to increase the audit frequency dramatically from annually to quarterly. Increasing auditing fourfold has not been justified and would substantially increase the demand for new auditors for

little, if any, gain. We expect the number of audits in California would increase from approximately 100 to 400 or more for PM2.5 alone. Annual audits for PM10 would be in addition to those for PM2.5. We have found that accuracy is directly tied to a reasonable frequency of calibrations and well-designed equipment. California would plan to continue annual audits.

#### Attachment--ARB Comments Sent to U.S. EPA in June 1996

## **FAX**

Russ Wiener, PhD ORD-USEPA RTP, NC

re: Nylon filter in FRM (PM2.5)

Dear Russ;

This is to follow up on the conversation between you, Bill Oslund and myself on our proposal to permit the use of a nylon filter backup with the upcoming PM2.5 FRM. Our purpose is to provide the option for states to obtain more "mass-conservative" measurements with the FRM without jeopardizing the FRM status. By allowing, as an option, an extension to the filter holder, one could add a nylon filter (and supporting screens if necessary) downstream of the primary Teflon filter to capture nitrate volatized from the primary filter. The secondary filters would be an option and not required. Its use would not, however, negate the sampler as an FRM. In addition to obtaining a more accurate measurement, it would provide you with a means of evaluating future samplers that address directly the question of nitrate retention of the filter. This would be especially important for equivalency testing and would allow you a vehicle for approving new devices that may yield higher results than the FRM.

Our recommendation stems from discussions with Dr. Walter John who successfully used the nylon filter as a backup to the Teflon filter in the "nitric acid shoot-out" in Claremont, CA in 1985. A full description of his approach using a dichotomous sampler is included in his paper (attached). A sketch is shown here.

Thank you for considering this modification. We believe it could be added to the FRM specifications with minimal design modifications. As you agreed, we would like to discuss this further within the week.

If you have any questions, please call either me (916.322.3726) or Bill Oslund (916.445.3745)

Sincerely,

(CO)

Jeff Cook

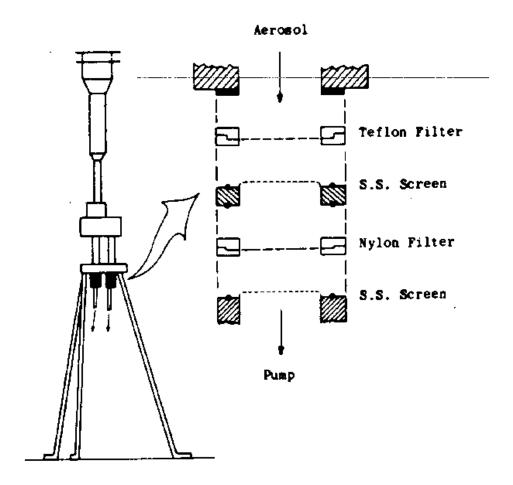


Fig. 1. Dichotomous sampler with filter holders modified to use a Teflon prefilter and a nylon after filter.